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धातु सम्मिश्र सिन्टेन — विशिष्टि

Indian Standard

METAL COMPLEX SYNTANS — SPECIFICATION

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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Price Group 4

Leather, Tanning Materials and Allied Products Sectional Committee, CHD 17

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Leather, Tanning Materials and Allied Products Sectional Committee had been approved by the Chemical Division Council.

Metal complex syntans are high molecular weight resinous products with ligating sites most suited for irreversible binding of the metals like Cr^{3+}, Al^{3+} and Zr^{4+}. These are homogeneous complex mixtures, which will have the advantage of uniform distribution inside the leather. Metal complex syntan is a major auxiliary used in the processing of leathers at the tanning and re-tanning stages.

The composition of the Committee responsible for formulation of this standard is given in Annex C.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard
METAL COMPLEX SYNTANS — SPECIFICATION

1 SCOPE

This standard prescribes requirements and the methods of sampling and test for metal complex syntans based on chromium (Cr^{3+}), aluminium (Al^{3+}) and zirconium (Zr^{4+}) intended for tanning and retanning purposes.

2 REFERENCE

The standard listed below contains provision, which through reference in this text constitutes provision of this standard. At the time of publication, the edition indicated was valid. All standards are subject to revisions and parties to agreements based on this standard is encouraged to investigate the possibility of applying the most recent edition of the standard indicated below:

IS No.	*Title*
1640 : 2007	Glossary of terms relating to hides, skins and leather (*first revision*)

3 TERMINOLOGY

For the purpose of this standard the definitions given in IS 1640 shall apply.

4 DESCRIPTION

Metal complex syntans shall in the form of a fine powder.

5 REQUIREMENTS

The metal complex syntans shall comply with the requirements given in Table 1 when tested according to the methods prescribed in Table 1.

Table 1 Requirement for Metal Complex Syntans

Sl No.	**Characteristic**	**Requirement**	**Method of Test, Ref to**	
			Clause of IS 13271	Annex
(1)	(2)	(3)	(4)	(5)
i)	Moisture content, percent by mass, *Max*	7	**7**	—
ii)	Total metal oxides, percent by mass, *Min*	10	—	A
iii)	Total ash content, percent by mass, *Max*	35	**16**	—
iv)	*p*H of 10 percent solution, *Min*	2.5	**13**	—

6 PACKING AND MARKING

6.1 Packing

The spray-dried powder or drum dried powder of the required metal complex syntans shall be packed in polythene-lined jute bags (moisture proof), paper or HDPE bags as agreed to between the purchaser and the supplier.

6.2 Marking

6.2.1 Packages shall be marked on the outside with the following information:

a) Name of material;
b) Net mass of the material;
c) Supplier's name or recognized trade-mark, if any; and
d) Batch No. and date of packing.

6.2.2 *BIS Certification Marking*

The bags containing metal complex syntans may also be marked with the Standard Mark.

6.2.2.1 The use of the Standard Mark is governed by the provisions of *Bureau of Indian Standards Act*, 1986 and Rules and Regulations made thereunder. The details of conditions under which the licence for use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

7 SAMPLING

7.1 Representative samples of the material shall be drawn in accordance with the method prescribed in Annex B and their criteria for conformity shall be judged in accordance with **7.3**.

7.2 Number of Tests

Each test sample shall be tested individually for all the requirements of this specification.

7.3 Criteria for Conformity

The lot shall be declared to conform to the requirements of this specification if the test sample passes all the tests.

ANNEX A

[*Table* 1, *Sl No.*(ii)]

DETERMINATION OF TOTAL METAL OXIDES

A-1 CHROMIUM CONTENT

Two different methods are described for the determination of chromium content in the metal syntan. The first method describes the titrimetric method (Method A) and the second method describes the determination of chromium as chromate by spectrophotometric technique (Method B).

A-1.1 Method A

Since the metal syntans contain substantial quantities of organic matter, it is necessary to pre-digest the material.

A-1.1.1 *Reagents*

A-1.1.1.1 *Concentrated sulphuric acid*

A-1.1.1.2 *Perchloric acid*, 60-70 percent solution.

A-1.1.1.3 *Concentrated nitric acid*

A-1.1.1.4 *Standard sodium thiosulphate solution* — 0.1N freshly standardized.

A-1.1.1.5 *Potassium iodide solution* — 10 percent (*w/v*).

A-1.1.1.6 *Starch indicator* — Triturate about 0.5 g of starch with 10 ml of water. Pour the resulting paste into 100 ml of boiling water, boil for 3 min, and allow the solution to cool.

A-1.1.2 *Procedure*

A-1.1.2.1 Weigh about 0.5 g of sample accurately and mix with sulphuric acid (3.5 ml), perchloric acid (11.5 ml) and nitric acid (5 ml) in a conical flask. Heat the mixture in a mantle placing a funnel at the mouth of the conical flask until the colour of the mixture turns orange red. Fumes of sulphur dioxide would be released just before the required colour change. Cool the flask and add 50 ml of water alongwith a few glass beads (anti-bumping). Continue heating for another 30 min to expel any free chlorine, if present. Cool the contents of the flask and make upto 100 ml in standard flask.

A-1.1.2.2 Pipette out 25 ml of the solution into a clean iodine flask and acidify with 10 ml of 1.0 N sulphuric acid. Immediately after acidification, add 10 ml of 10 percent potassium iodide solution with intermittent shaking. Titrate the liberated iodine with standard sodium thiosulphate solution using starch indicator. Starch indicator is to be added towards the end of the titration. The end point is the disappearance of the blue colour. Typical chrome content in the syntan samples is expressed as Cr_2O_3.

A-1.1.3 *Calculation*

A-1.1.3.1 Chromium content, as Cr_2O_3, in the syntan is calculated using the factor

1 ml of 1 N titrant = 0.025 3 g of Cr_2O_3.

$$\text{Chromium content, as } Cr_2O_3 \text{, percent by mass} = \frac{V \times N \times 0.025\,3 \times 100 \times 4}{M}$$

where

V = volume of sodium thiosulphate consumed,

N = normality of thiosulphate used, and

M = mass of the sample taken.

A-1.2 Method B

A-1.2.1 *Reagents*

a) *Sodium hydroxide* — Reagent grade, and

b) *Sodium hydroxide solution* — 0.1 N.

A-1.2.2 *Equipment*

A-1.2.2.1 *Spectrophotometer*, with least count of 0.1nm.

A-1.2.3 *Procedure*

A-1.2.3.1 Pipette out 2 ml of the pre-digested solution, obtained at **A-1.1.2.1**, into a 25 ml volumetric flask. Add a few pellets of solid sodium hydroxide to make the solution distinctly alkaline. Cool the contents and make the volume up to the mark with double distilled water. Measure the optical density at 372 nm using 0.1 N sodium hydroxide solution as blank. Estimate the concentration of chromium in the sample using a molar absorptivity of 4.82×10^3 M^{-1} cm^{-1} at 372 nm when measured as chromate.

A-1.2.4 *Calculation*

$$\text{Chromium content as } Cr_2O_3 \text{, percent by mass} = \frac{OD \times 25 \times 100 \times 52 \times 100 \times 1.46}{4\,820 \times 2 \times 1\,000 \times M}$$

where

OD = optical density, and

M = mass of the sample taken.

A-2 DETERMINATION OF ALUMINIUM

A-2.1 Reagents

A-2.1.1 *Mixed Indicator* — Mix 3 parts of 1 percent aqueous solution of thymol blue with 1 part of 1percent aqueous solution of cresol red.

A-2.1.2 *Potassium Hydroxide Solution* — 5.0 N and 0.2 N.

A-2.1.3 *Potassium Tartarate* — reagent grade.

A-2.1.4 *Potassium Fluoride* — 20 percent solution (*w/v*).

A-2.1.5 *Standard Hydrochloric Acid* — 0.1 N.

A-2.2 Equipment

A-2.2.1 *p*H *meter*, with least count of 0.1.

A-2.3 Procedure

A-2.3.1 Digested samples of the syntans as used for chromium determination (*see* **A-1.1.2.1**) may also be employed for the determination of aluminium content.

A-2.3.2 Add a few drops of mixed indicator to the 25 ml of the solution. When the colour of the solution turns red, add suitable quantities of 5.0 N potassium hydroxide until the red colour disappears. Cool the contents of the flask and add 3 g of potassium tartarate for every 10 ml of the solution. Using 0.2 N potassium hydroxide adjust the *p*H of the solution to a *p*H of 8.3 (use a *p*H meter). Add 20 ml of potassium fluoride solution. This will result in increase in *p*H of the solution. Titrate the contents against 0.1 N hydrochloric acid to a *p*H of 8.3.

A-2.4 *Calculation*

A-2.4.1 The aluminium content as percentage by mass in the syntan is calculated using the factor 0.017.

(1 ml of 1 N hydrochloric acid = 0.017 g of Al_2O_3).

$$\text{Aluminium content, as } Al_2O_3\text{, percent by mass} = \frac{V \times N \times 0.017 \times 100 \times 4}{M}$$

where

V = volume of HCl consumed,

N = normality of HCl used, and

M = mass of the sample taken for estimation.

A-3 DETERMINATION OF ZIRCONIUM

Zirconium may be estimated by two methods, namely, Method A and Method B.

A-3.1 Method A (Gravimetric Method)

A-3.1.1 *Principle* — Zirconium is precipitated from hydrochloric acid solution with Mandelic acid as zirconium mandelate, which is ignited and weighed as the zirconium dioxide.

A-3.1.2 *Reagents*

A-3.1.2.1 *Fusion mixture* — Mix equal parts of potassium carbonate and sodium carbonate.

A-3.1.2.2 *Sodium peroxide* — Analytical grade.

A-3.1.2.3 *Concentrated hydrochloric acid*

A-3.1.2.4 *Mandelic acid* — 16 percent aqueous solution (*m*/*v*).

A-3.1.3 *Equipment*

A-3.1.3.1 *Platinum crucible*

A-3.1.4 *Procedure*

A-3.1.4.1 Mix intimately one gram of metal complex syntan with 3 to 4 times its mass of the fusion mixture (*see* **A-3.1.2.1**). Transfer the mixture to a platinum crucible and ignite, gently at first and then vigorously, ensuring that there is a generous supply of air. When the process is complete, add a small portion of sodium peroxide. Heat the mixture to fusion, for 10 to 15 min. Cool the melt. Extract with boiling water and transfer quantitatively into a 250 ml beaker. Add about 20 ml of concentrated hydrochloric acid to the above extract. Boil the mixture till carbon dioxide is expelled. Make up the clear solution to 100 ml using double distilled water.

A-3.1.4.2 Pipette 20 ml of the above stock solution into a clean beaker. Add about 50 ml of 16 percent aqueous Mandelic acid solution and dilute to 100 ml. Raise the temperature of the solution to 85°C and maintain for 20 min. Filter the resulting precipitate using an ashless filter paper (like Whatman filter paper No. 42), and wash with a hot solution containing 2 percent Hydrochloric acid and 5 percent Mandelic acid. Transfer the precipitate along with the filter paper to a pre-weighed platinum crucible and ignite gently at first and then vigorously ensuring that there is a generous supply of air, at a temperature of 900-1 000°C. Weigh the residue after cooling.

A-3.1.5 *Calculation*

A-3.1.5.1 The zirconium content as percentage by mass in the syntan is calculated as follows:

$$\text{Zirconium content as } ZrO_2\text{, percent by mass} = \frac{(B - A) \times 100 \times 5}{M}$$

where

A = weight of empty crucible,

B = weight of crucible + residue, and

M = mass of the sample taken.

A-3.2 Method B (Spectrophotometric Method)

This procedure is intended for determining zirconium, when present in small amounts, as the 8-hydroxyquinolinate in chloroform.

A-3.2.1 *Reagents*

A-3.2.1.1 *Zirconium standard* (*40 µg/ml*) — Dissolve exactly 0.141 3 g of analytical grade zirconium oxychloride octahydrate in 50 ml of 1 : 1 perchloric acid. Dilute to 1 litre with 1 : 1 perchloric acid.

A-3.2.1.2 *Ammonium chloride — ammonium tartrate solution* — Dissolve 120 g of ammonium chloride and 120 g of ammonium tartrate in water and dilute to 1 litre.

A-3.2.1.3 *Dilute hydrofluoric acid* — Dilute 48 percent hydrofluoric acid 1:10 and store in polyethylene.

A-3.2.1.4 *8-hydroxyquinoline, 5 percent solution* — Dissolve 5 g of 8-hydroxyquinoline in 100 ml acetone.

A-3.2.2 *Procedure*

A-3.2.2.1 Mix 0.5 g of the sample with 3.5 ml of sulphuric acid, 11.5 ml of perchloric acid and 5 ml of nitric acid in a conical flask. Heat the mixture in a mantle placing a funnel at the mouth of the conical flask until a colourless, clear solution is obtained. It can be seen that fumes of sulphur dioxide would be released just before the completion of the digestion. Cool the flask and add 50 ml of water. Continue the heating for another 30 min to expel any chlorine, if present. Make up the contents of the flask upto 100 ml with double distilled water.

A-3.2.2.2 Transfer 5 ml of the above stock solution to a 100 ml beaker. Add about 10 ml of dilute hydrofluoric acid and 10 ml of ammonium chloride-ammonium tartrate solution and dilute the mixture to 50 ml with double distilled water. Adjust the *p*H of the solution to 8.9 ± 0.1 with ammonia. Add 1 ml of 5 percent 8-hydroxyquinoline solution and allow the mixture to stand for 30 min. Transfer the mixture to a 125 ml separating funnel and extract with three 10 ml portions of chloroform, shaking for 30 s each time. Collect the chloroform extracts quantitatively in a 50-ml volumetric flask and make upto the mark with chloroform. Add one gram of anhydrous sodium sulphate to the flask to remove any trace of water. Carry out a reagent blank also. Measure the absorbance of the extract using a spectrophotometer at 386 nm, using the reagent blank as the reference. The amount of zirconium is determined from a calibration graph prepared previously from standard zirconium solution.

A-3.2.2.3 *Calculation*

A-3.2.2.3.1 The zirconium content as percentage by mass in the syntan is calculated as follows:

$$\text{Zirconium content, as } ZrO_2\text{, percent by mass} = \frac{A \times 20 \times 100 \times 1.35 \times 1\,000}{M}$$

where

A = amount of zirconium from calibration graph, in μg/ml; and

M = mass of the sample taken.

A-4 TOTAL METALLIC OXIDES

This is obtained by summing up the quantities of chromium content, as Cr_2O_3, aluminium content, as Al_2O_3, and zirconium content as oxide of zirconium.

A-5 DETERMINATION OF CHROMIUM, ALUMINIUM AND ZIRCONIUM BY ATOMIC ABSORPTION SPECTROMETRY (AAS)

A-5.1 General

Combustion flames convert inorganic analytes in solution into free atoms. If light of resonance wavelength is passed through these free atoms, part of the light is absorbed and the extent of absorption is proportional to the number of ground state atoms present in the flame.

A-5.2 Reagents

A-5.2.1 *Concentrated Hydrochloric Acid*

A-5.2.2 *Concentrated Nitric Acid*

A-5.2.3 *Hydrofluoric Acid*

A-5.2.4 *Preparation of Calibration Graph*

A-5.2.4.1 Prepare a stock solution of the metal (Cr, Al, Zr) by dissolving 1 g of pure metal in 10 ml of hydrochloric acid and 10 ml of nitric acid. Heat gently until no more fumes of nitric oxide are obtained. Cool, transfer the solution quantitatively into a plastic beaker and add 5 ml of hydrofluoric acid, ensuring the temperature does not rise above 30°C. Transfer this solution to 100 ml plastic graduated flask and make upto the volume with deionized water. Appropriate dilution of suitable aliquots of the stock solution can be used to give standards containing 100, 200, 300, 400 and 500 μg/ml of the metal.

A-5.2.4.2 Atomize aliquots of standard solutions under conditions given in Table 2 and read the absorbance.

A-5.2.4.3 Plot absorbance against concentration of the metal, in μg/ml.

A-5.3 Procedure

Weigh accurately 0.5 g of the metal complex syntan and digest the sample in the same manner as described in **A-5.2.4**. Atomise aliquots of the test solution. Read the concentration of the metal (in µg/ml) from the calibration graph.

A-5.4 Calculation

A-5.4.1 Estimate the individual metals using the formula,

$$\text{Metal, (as Al, Cr, Zr), percent by mass} = \frac{M_1 \times 100}{M_2}$$

where

M_1 = mass of metal found in sample solution, in g; and

M_2 = mass of the metal complex syntan taken for the test, in g.

A-5.4.2 The sum of the individual metals gives the total metallic content in the syntan.

A-5.4.3 Total metal oxide, as percent by mass, is obtained by using the following formula:

$$X \times 1.46 + Y \times 1.86 + Z \times 1.35$$

where

X = chromium content,

Y = aluminium content, and

Z = zirconium content.

Table 2 AAS Conditions

(*Clause* A-5.2.4.2)

Sl No. (1)	Element (2)	Wavelength, λ nm (3)	Flame (4)	Conc Range µg/ml (5)
i)	Aluminium	309.3	NA	40-200
ii)	Chromium	357.9	AA	2-8
iii)	Zirconium	360.1	NA	200-500

NA = Mitrous oxide/acetylene.

AA = air/acetylene.

ANNEX B

(*Clause* 7.1)

SAMPLING

B-1 SCALE OF SAMPLING

B-1.1 Batch

In a single batch, all the metal complex syntans (spray dried or drum dried) prepared and dried under similar conditions shall constitute one batch.

B-1.2 For ascertaining the conformity of the material to the requirements of this specification, each batch shall be considered separately.

B-1.3 A random sample would be drawn for testing.

B-2 PREPARATION OF SAMPLE

B-2.1 Test Sample

The material obtained from each batch shall be divided into 3 equal parts, each forming a test sample representing the batch. One set of test sample shall be marked for the purchaser, another set for the supplier and the third set as a referee sample.

B-2.2 All the test samples shall be immediately transferred to separate sample containers and shall be sealed air-tight and marked with full particulars necessary for proper identification; such as the name of the supplier, the place and year of production, batch number, date of sampling and the name of the sample.

B-2.3 Referee Sample

The referee sample shall bear the seal of both the supplier and the purchaser and shall be kept at a place and under conditions agreed to between the two for use in the case of dispute.

ANNEX C

(*Foreword*)

COMMITTEE COMPOSITION

Leather, Tanning Materials and Allied Products Sectional Committee , CHD 17

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Central Leather Research Institute, Chennai	Director (***Chairman***) Dr S. Sadulla (*Alternate* I) Dr C. Muralidharan (*Alternate* II)
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BUREAU OF INDIAN STANDARDS

Headquarters :

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110 002
Telephones : 2323 0131, 2323 3375, 2323 9402

Telegrams : Manaksanstha
(Common to all offices)

Regional Offices :

Regional Offices	Address	Telephone
Central	Manak Bhavan, 9 Bahadur Shah Zafar Marg NEW DELHI 110 002	2323 7617 2323 3841
Eastern	1/14 C.I.T. Scheme VII M, V. I. P. Road, Kankurgachi KOLKATA 700 054	2337 8499, 2337 8561 2337 8626, 2337 9120
Northern	SCO 335-336, Sector 34-A, CHANDIGARH 160 022	260 3843 260 9285
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